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(71)Applicant : SHIN ETSU CHEM CO LTD

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(72)Inventor : OHASHI TAKESHI
NOMURA TADAO

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(54) RARE EARTH/IRON/BORON MAGNET AND MANUFACTURE THEREOF

(57)Abstract:

PROBLEM TO BE SOLVED: To directly make a rare earth/boron/iron magnet anisotropic and in bulk in the presence of a liq. phase by the hot uniaxial deforming from its quenched thin tape.

SOLUTION: A nanocomposite magnet is composed of R (one or more kinds of rare earth elements including Y), Fe (or Fe substituted by a specified quantity of Co), B and, if necessary, M (one or more kinds of Al, V, Mo, Zr, Ti, Sn, Cu, and Ga 4 atm.% or more), the Fe content is 82 atm.% or more, and the hard phase is of R₂Fe₁₄B and the soft phase is of Fe₃B. A rare earth/iron/boron magnet is a nanocomposite magnet made anisotropic and in bulk which has a hard phase of R₂Fe₁₄B exchange-bonded with a soft phase having a higher saturation magnetization than the hard phase and is obtained by the direct hot uniaxial deforming, quick temp. rise and quenching process from a quenched thin tape having a compsn. wherein a liq. phase exists in the hot uniaxial deforming process.

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(21) 出願番号	特願平11-357402	(71) 出願人	000002060 信越化学工業株式会社 東京都千代田区大手町二丁目6番1号
(22) 出願日	平成11年12月16日 (1999. 12. 16)	(72) 発明者	大橋 健 福井県武生市北府2丁目1番5号 信越化学工業株式会社磁性材料研究所内
(31) 優先権主張番号	特願平10-358573	(72) 発明者	野村 忠雄 福井県武生市北府2丁目1番5号 信越化学工業株式会社磁性材料研究所内
(32) 優先日	平成10年12月17日 (1998. 12. 17)	(74) 代理人	100062823 弁理士 山本 亮一 (外2名)
(33) 優先権主張国	日本 (J P)		

(54) 【発明の名称】 希土類・鉄・ボロン系磁石とその製造方法

(57) 【要約】

【課題】 $R_2Fe_{1-x}B$ をハード相としたナノコンポジット磁石であって、異方性化かつバルク化した希土類・鉄・ボロン系磁石及びその製造方法を提供する。

【解決手段】 希土類・鉄・ボロン系合金の急冷薄帯から温間一軸変形により、液相の存在下で、直接、異方性化する。

【特許請求の範囲】

【請求項1】 R (Yを含む希土類元素の1種以上)、Fe、Bよりなり、Feの比率が82原子%以上で、ハード相が $R_2Fe_{14}B$ 、ソフト相がFe又は Fe_2B であるナノコンポジット磁石であって、希土類・鉄・ボロン系合金の急冷薄帯から温間一軸変形により、液相の存在下で、直接、異方性化して得られることを特徴とする希土類・鉄・ボロン系磁石。

【請求項2】 R (Yを含む希土類元素の1種以上)、Fe、Co、Bよりなり、FeとCoの合計した比率が82原子%以上、Coの比率がFeとCoの合計の20原子%以内で、ハード相が $R_2(Fe-Co)_{14}B$ 、ソフト相が $Fe-Co$ 又は $(Fe-Co)_2B$ であるナノコンポジット磁石であって、希土類・鉄・ボロン系合金の急冷薄帯から温間一軸変形により、液相の存在下で、直接、異方性化して得られることを特徴とする希土類・鉄・ボロン系磁石。

【請求項3】 R (Yを含む希土類元素の1種以上)、Fe、M (Al、V、Mo、Zr、Ti、Sn、Cu、Gaの1種以上、原子百分比で4%以内)、Bよりなり、Feの比率が82原子%以上、基本的にハード相が $R_2Fe_{14}B$ 、ソフト相がFe又は Fe_2B であるナノコンポジット磁石であって、希土類・鉄・ボロン系合金の急冷薄帯から温間一軸変形により、液相の存在下で、直接、異方性化して得られることを特徴とする希土類・鉄・ボロン系磁石。

【請求項4】 R (Yを含む希土類元素の1種以上)、Fe、Co、M (Al、V、Mo、Zr、Ti、Sn、Cu、Gaの1種以上、原子百分比で4%以内)、Bよりなり、FeとCoの合計した比率が82原子%以上、Coの比率がFeとCoの合計の20原子%以内で、基本的にハード相が $R_2(Fe-Co)_{14}B$ 、ソフト相が $(Fe-Co)$ 又は $(Fe-Co)_2B$ であるナノコンポジット磁石であって、希土類・鉄・ボロン系合金の急冷薄帯から温間一軸変形により、液相の存在下で、直接、異方性化して得られることを特徴とする希土類・鉄・ボロン系磁石。

【請求項5】 希土類・鉄・ボロン系合金の急冷薄帯を、液相の存在下で、温間一軸変形して、直接、異方性化することを特徴とする請求項1～4記載の希土類・鉄・ボロン系磁石の製造方法。

【請求項6】 温間一軸変形後に磁石合金周縁部に濃縮される液相を除去することを特徴とする請求項5記載の希土類・鉄・ボロン系磁石の製造方法。

【請求項7】 温間一軸変形過程において、保持温度までの昇温を2秒以上5分以内に行い、かつ、保持温度から300℃以下までの降温を5秒以上10分以内に行うことを特徴とする請求項5記載の希土類・鉄・ボロン系磁石の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、電子機器、特にハードディスクドライブのヘッド駆動用アクチュエータに使用すると最適な希土類・鉄・ボロン系磁石及びその製造方法に関する。

【0002】

【従来の技術】近年、佐川、Croaたちにより、Nd、 $Fe_{14}B$ を主相とするネオジム・鉄・ボロン磁石が発見されて以来、ネオジム・鉄・ボロン系磁石は、その組成や添加物の最適化及び高い磁気特性を引き出す製造方法の開発、改良等により、その磁気特性を改善してきた。各種製造方法のうち、粉末冶金法で作られるネオジム・鉄・ボロン系焼結磁石は、化学量論比に近づける低Nd高Fe組成化（なお、 $Nd_2Fe_{14}B$ 化合物の原子百分比は、11.8%Nd、5.9%B、82.3%Fe）、低酸化プロセス、高磁場配向、組織微細化等の組合せにより、最大エネルギー積 $(BH)_{max}$ の理論値の約88%に相当する $(BH)_{max}$ が実現されている。しかしながら、粉末冶金法のプロセスや組成の改良等による磁気特性の改善も限界に達しつつある。具体的には、粉末冶金法では、化学量論比組成を超えるFe組成（82%Fe以上）をもつ高性能のネオジム・鉄・ボロン系磁石を得ることができない。その理由は、高Fe組成になると必然的にFe相が生じ、ソフト磁性を有するこのFe相が磁化反転の原因となって保磁力が得られなくなるからであり、また、化学量論比よりNdリッチな組成（つまりFeブアな組成）で存在する低融点Ndリッチ相が焼結過程で液相となり、 $Nd_2Fe_{14}B$ 粒子の表面をクリーニングすることにより、核発生成長型の保磁力発生に寄与していると考えられているからである。

【0003】異方性ネオジム・鉄・ボロン系磁石の製造方法として、粉末冶金法以外には温間一軸変形法が知られている。この方法は、アモルファス薄帯の熱処理あるいは冷却速度の制御により得られたネオジム・鉄・ボロンの微結晶薄帯である急冷薄帯（商品名MQ1、MQ1社製）をホットプレスでバルク化して、バルク等方性磁石（商品名MQ2、MQ1社製）とし、該バルク等方性磁石を温間一軸変形することにより、加圧方向に容易磁化軸が配向した異方性ネオジム・鉄・ボロン磁石（商品名MQ3、MQ1社製）を得る方法である。温間一軸変形法により得られるネオジム・鉄・ボロン磁石の異方性の程度は、温間加圧変形の程度と相関があり、変形度が大きくなるほど異方性の度合も大きくなる。そして、この方法により、 $(BH)_{max}$ の理論値の約75%に相当する $(BH)_{max}$ が実現されている。しかしながら、温間一軸変形法は、Ndリッチな低融点相（温間一軸変形過程では液相）が存在する組成でしか変形が起こらないため、低融点相が存在しない化学量論比組成や、より高いFe組成の場合は適用できない。したがって、

従来の温間一軸変形法では、粉末冶金法で得られるネオジム・鉄・ボロン系磁石よりも磁気特性を向上させることは困難であった。

【0004】ソフト相とハード相が微細組織(10nmオーダー)を形成するナノコンポジット磁石は、ソフト相とハード相が交換結合により一体化されている。そして、このナノコンポジット磁石は、ソフト相が存在しているにもかかわらず永久磁石特性を示すことが、シミュレーションと実測の両方で実証されている。そのため、上記ソフト相に飽和磁化の高い材料を使用すれば、高い飽和磁化と十分な保磁力を備え、ハード相を超える高い磁気特性を示す可能性がある。ナノコンポジット磁石では、ソフト相(Fe、FeCo、Fe₂B・FeN系化合物等)とハード相(Nd₂Fe₁₄B、SmCo₅、Sm₂Co₁₇、Sm₂Fe₁₇Nx、NdTiFe₁₁Nx、その他の窒化物)は、特定の組合せに限定される必然性はなく、括弧内のものを自由に組合せることが可能であり、また、ハード相の組成により常に限定されるわけではない。

【0005】しかしながら、ナノコンポジット磁石は、粒子間の交換結合が有効な10nmオーダーの微細組織においてのみ存在できるものであるが、このような微細組織を異方性化することは実現していない。ナノコンポジット磁石の特徴は、等方性組織であっても、ソフト相が存在することにより、比較的高いBr(残留磁束密度)を示す点にあるが、等方性組織では十分な保磁力、高い(BH)maxを発現することができない。

【0006】また、ナノコンポジット磁石には、バルク化できないという問題点もある。ナノコンポジット磁石は、通常、液体急冷法やメカニカルアロイング法等により作製され、粉末又は薄帯の形で得られるが、得られたナノコンポジット組織を肥大化させずに、バルク磁石とする方法はまだ考案されていない。パルス超高压力により粉体をバルク化させる特殊な方法はあるものの、到底実用化に適した方法ではない。以上のように、ナノコンポジット磁石において、異方性化とバルク化を同時に実現することは不可能であった。

【0007】

【発明が解決しようとする課題】そこで、本発明は、R₂Fe₁₄Bをハード相としたナノコンポジット磁石であって、異方性化かつバルク化した希土類・鉄・ボロン系磁石及びその製造方法を提供することを目的とする。

【0008】

【課題を解決するための手段】本発明は、R(Yを含む希土類元素の1種以上)、Fe(又は所定量のCoで置換したFe)、B、さらには必要によりM(Al、V、Mo、Zr、Ti、Sn、Cu、Gaの1種以上、原子百分比で4%以内)よりなり、Feの比率が82原子%以上で、ハード相がR₂Fe₁₄B、ソフト相がFe又はFe₂Bであるナノコンポジット磁石であって、希土類

・鉄・ボロン系合金の急冷薄帯から温間一軸変形により、液相の存在下で、直接、異方性化して得られることを特徴とする希土類・鉄・ボロン系磁石である。別の本発明は、希土類・鉄・ボロン系合金の急冷薄帯を、液相の存在下で、温間一軸変形して、直接、異方性化することを特徴とする上記希土類・鉄・ボロン系磁石の製造方法である。この場合、液相は、ハード相に対して濡れ性のあるLa-Fe系又はR-Cu系の低融点合金からなるのが好ましい。本発明では、温間一軸変形後に磁石合金周縁部に濃縮される液相を除去し、また、温間一軸変形過程において、保持温度までの昇温は2秒以上5分以内に行い、かつ、保持温度から300℃以下までの降温は5秒以上10分以内に行うのがよい。

【0009】

【発明の実施の形態】本発明の希土類・鉄・ボロン系磁石は、R₂Fe₁₄Bをハード相とし、該ハード相より飽和磁化の高いソフト相との間で交換結合させ、異方性化かつバルク化したナノコンポジット磁石であり、温間一軸変形過程において、液相が存在する組成で急冷薄帯からの直接温間一軸変形、急速昇温・急速冷却のプロセスにより得られるものである。

【0010】本発明の希土類・鉄・ボロン系磁石は、R(Yを含む希土類元素の1種以上)、Fe(又はFe-Co)、B、さらには必要によりM(Al、V、Mo、Zr、Ti、Sn、Cu、Gaの1種以上)よりなり、かつFe(又はFe-Co)の比率は、R₂Fe₁₄Bの化学量論比よりFeリッチな82原子%以上の組成をもつナノコンポジット磁石であり、そのハード相はR₂Fe₁₄B(又はR₂(Fe-Co)₁₄B)、ソフト相はFe(又はFe-Co)又はFe₂B(又は(Fe-Co)₂B)からなる。RはYを含む希土類元素であるが、Nd、Prを主体とし、保磁力を増大させるため、Rの一部にTbやDyを使用すると効果的である。また、上記FeはCoで置換可能であるが、Co置換によりハード相の磁気特性が低下するので、その置換比率は原子百分比でFeとCoの合計の20%を上限とするのが望ましい。さらに、Fe又はFe-Coは添加元素M(Al、V、Mo、Zr、Ti、Sn、Cu、Gaの1種以上)で少量、置換してもよい。Mは組織の微細化に効果があり、保磁力増大に寄与するものの、Mによる置換の程度が原子百分比で全組成に対し4%以上になると磁気特性が低下しすぎることになるので、4%以内とするのが望ましい。ただし、Mの添加量が少なすぎると微細化の効果が現れなくなるので、好ましくは0.1%以上添加した方がよい。なお、希土類・鉄・ボロン系合金の作製や磁石製造の工程で混入する、C、N、Oその他の不純物の量については少ないほど望ましいが、1%以内とすることは不可避である。

【0011】本発明の希土類・鉄・ボロン系磁石は、液体急冷法又はメカニカルアロイング法で得られる、薄帯

あるいは粉末（以下、これらを急冷薄帯と言う）から製造される。急冷薄帯は粒子間に交換結合が生じた10nmオーダーの微細組織状態又はアモルファス状態のどちらでもよいが、温間一軸変形の過程で磁石合金微粒子の粒径の肥大を極力抑制するためにはアモルファス状態の方が望ましい。

【0012】急冷薄帯を温間一軸変形して直接、異方性化することにより、本発明の希土類・鉄・ボロン系磁石が得られる。従来方法においては、急冷薄帯を温間一軸変形して異方性化する場合、一度ホットプレスにより急冷薄帯をバルク化し、得られた等方性磁石ブロックを、もう一度昇温した後、温間一軸変形して異方性化していた。しかしながら、本発明では、急冷薄帯を急速昇温して、直接、温間一軸変形する。そのため、異方性化過程では急冷薄帯に対して必要最小限の熱処理しか行われないので、温間一軸変形過程における微細組織の肥大化が抑制され、その結果、粒子間の交換結合が十分に行われることになる。

【0013】〔従来の技術〕で述べたように、Feの比率が82原子%以上で、ハード相が $R_2Fe_{14}B$ である希土類・鉄・ボロン系合金は、温間で一軸加圧を行っても、ほとんど変形が起ころず異方性化しない。そこで、この問題を解決するため、本発明では、希土類・鉄・ボロン系磁石合金を、Feの比率が82原子%以上で、かつ、温間一軸変形の過程で液相が存在するように調製する。この液相は希土類・鉄・ボロン系磁石合金と全く関連性のないもの（例えば、低融点半田合金）では、希土類・鉄・ボロン系磁石合金との濡れ性が悪く、温間一軸変形に寄与しないので、温間一軸変形過程で液相となり、かつ、希土類・鉄・ボロン系磁石合金と濡れ性を有するものが必要となる。本発明者らは、このような条件を満たす合金について種々検討した結果、La-Fe系合金あるいはR-Cu系合金が好ましいことを見出した。このような組織を得るには、組織中にLa、Cuを直接添加するか、La-Fe系合金、R-Cu系合金をR-Fe-B系合金に配合させる。この場合、La、Cuの添加量は、当初の組成に対し2wt%以下にすることが好ましい。なお、La-Fe系の二元合金は金属間化合物を作らず、また、La-Fe-B系の三元合金は、 $La_2Fe_{14}B$ を非常に生成しにくい。La-Fe系の二元合金は、Laリッチ側で共晶反応により800℃以下で液相となる。この液相は、元の希土類・鉄・ボロン系磁石合金と類似性があり $R_2Fe_{14}B$ 相との濡れ性が良好であるので、温間一軸変形に寄与する。一方のR-Cu系合金も、Cuは $R_2Fe_{14}B$ のFeを置換せず、かつR-Cu系で生成される化合物は融点が高いため、本発明の液相として適当である。本発明では、このような低融点相が温間一軸変形過程で液相となり、該液相を介して再配向を行うことにより、従来技術では困難であった異方性化が達成される。

【0014】液相を媒介とした温間一軸変形により、急冷薄帯を十分に加圧変形して直接、異方性化した後は、La-Fe系（又はR-Cu系）の低融点合金からなる液相は、圧力がフリーとなる磁石合金周縁部に絞り出されて濃縮される。その際、磁石合金中心部には液相成分はほとんど存在しない状態となる。従来方法のように、磁石合金を等方性バルク磁石にした後に異方性化する方法では、低融点液相成分は磁石合金周縁部で濃縮されず、内部に均一に分散する。すなわち、低融点液相が磁石合金周縁部で濃縮される現象は、本発明のように、急冷薄帯を直接、温間一軸変形した場合にのみ見られる。低融点液相は非磁性であるため永久磁石の磁気特性には寄与せず、温間一軸変形が終了した後は磁気特性を低下させるのみである。したがって、低融点液相が濃縮された磁石合金周縁部を除去して、ほとんど磁性相よりなる中心部を使用することにより、高い磁気特性を有する異方性ナノコンポジット磁石が得られる。

【0015】急冷薄帯の温間一軸変形は、短時間で行わないと微細粒子の肥大化が起こるので、これを避けるために、温間一軸変形過程において、温間一軸変形させる保持温度までの昇温を2秒以上5分以内に行い、かつ、保持温度から300℃以下までの降温は5秒以上10分以内に行うことが好ましい。この場合、保持温度は500～1000℃である。このような短時間の高速昇温と降温が可能な具体的方法の一例として、通電粉末圧延法がある。この方法は、図1に示したように機械粉碎による急冷薄帯の粉末1をホッパー2上部よりロール3内に投入し、被圧延粉末を介して大電流を通電するとロール出口が最高温度となるので、このロール3で粉末1を加圧することによりシート4に成形し、一軸温間変形を行う方法である。この方法によれば加圧する場合、ロール3の軸に平行な方向は圧力が逃げるため一軸圧縮となる。また、ロール3による圧縮が始まるまでは、急冷薄帯は粉末状であるため、通電しても電源5からは電流が流れず、したがって、ほとんど昇温が起こらない。すなわち、ロール3内において、ある程度圧縮されることによって初めて通電し、また、圧延材がロール3を出た時点で通電しなくなって降温フェーズに入るため、急冷薄帯が加熱されている時間は極めて短時間である。

【0016】この通電粉末圧延法によれば、アモルファス状態（又は微細結晶状態）から結晶化した組織になるまでに、磁石合金の粒径はあまり肥大せず、10nmオーダーの微細組織のままの異方性バルク化ナノコンポジット磁石が得られる。温間一軸変形を行う最高温度と昇温・降温の速度は、ロール3間に通電する電流値とロール3回転数を調節し、加圧変形度合は、ロール間の圧力と間隔を調節することで最適化することができる。通電圧延部は圧延体の酸化劣化を防止するため、不活性ガス雰囲気又は真空雰囲気であることが望ましい。ロール3は1段でも多段でもよい。なお、温間一軸変形を行う方

法は、上記方法に限定されるものではなく、加圧放電焼結法等の同様な機能を有する方法であれば、いずれでもよい。

【0017】

【実施例】以下、本発明の実施例を示すが、本発明はこれに限定されるものではない。

（実施例1）原子百分比で8%Nd、1%La、76%Fe、10%Co、5%B及び不可避の不純物よりなる溶解合金を、減圧Arガス雰囲気下で液体急冷を行い、周速60m/secの単ロール装置でアモルファス急冷薄帯とした。次に、該アモルファス急冷薄帯を機械で粉砕して、100メッシュ以下の粉末とし、Arガス雰囲気内で、通電粉末圧延法により、得られた粉末の異方性化及びバルク薄板化を同時に行った。その際、一軸圧は平均で500kg/cm²で、電流は10kA、ロール周速は1mm/secであり、温間一軸変形させる保持温度領域（800℃）まで約20秒で室温から昇温し、約40秒で300℃以下まで降温した。作製された20mm幅で1mm厚の薄板のうち、Laの大部分が縁部に濃縮されており、該部分である両端2.5mmを除去して、残り15mm幅の薄板を加圧方向にBr、iHcを計測したところ、Br=1.66T、iHc=800kA/mであり、異方性化した磁気特性が得られた。また、得られた薄板の組成は、8.1%Nd、0.1%La、76.5%Fe、10.1%Co、5.1%Bであり、Fe-CoとNd₂(Fe-Co)₃Bよりなっていた。

【0018】（実施例2）原子百分比で6%Pr、1.5%La、87.5%Fe、5%B及び不可避の不純物よりなる溶解合金を、実施例1と同じ条件で急冷してアモルファス急冷薄帯を作製した。また、該条件と同じ条件の通電粉末圧延法でバルク薄板とし、作製された20mm幅で1mm厚の薄板のうち、縁部に濃縮されていたLaの部分である両端2.5mmを除去して、残り15mm幅の薄板を加圧方向（1mm厚方向）にBr、iHcを計測したところ、Br=1.53T、iHc=990kA/mであり、異方性化した磁気特性が得られた。また、得られた薄板の組成は、6.5%Pr、0.1%La、88%Fe、5.4%Bで、FeとPr₂Fe₁₄Bよりなっていた。

【0019】（実施例3）原子百分比で8%Nd、8

2.5%Fe、8%B、1%Al、0.5%Ti及び不可避の不純物よりなる予め合金化したものを重量比で95%と、SmCu合金を重量比で5%を所定比に混合し、該混合溶解合金を減圧Arガス雰囲気下で液体急冷を行い、周速60m/secの単ロール装置でアモルファス急冷薄帯とした。次に、通電粉末圧延の電流値を15kAとした以外は、実施例1と同様にして縁部に濃縮していたCuを除去して薄板を作製し、加圧方向にBr、iHcを計測したところ、Br=1.45T、iHc=1250kA/mであり、異方性化した磁気特性が得られた。得られた薄板を手動粉砕した20メッシュ以下の粉末をX線回折で測定したところ、基本的にNd₂Fe₁₄BとFe₃Bからなることが確認できた。

【0020】（実施例4）原子百分比で8%Nd、1%La、84.5%Fe、5%B、1%Cu、0.5%Mn及び不可避の不純物よりなる溶解合金を減圧Arガス雰囲気下で液体急冷を行い、周速60m/secの単ロール装置でアモルファス急冷薄帯とした。次に、通電粉末圧延の電流値を15kAとした以外は、実施例1と同様にして縁部に濃縮していたCuを除去して薄板を作製し、加圧方向にBr、iHcを計測したところ、Br=1.53T、iHc=1040kA/mであり、異方性化した磁気特性が得られた。得られた薄板を手動粉砕した20メッシュ以下の粉末をX線回折で測定したところ、基本的にNd₂Fe₁₄BとFe₃Bからなることが確認できた。

【0021】

【発明の効果】本発明により、希土類・鉄・ボロン系磁石合金を、液相の存在下、高速昇温、高速降温条件で直接、温間一軸変形することにより、今までナノコンポジット磁石で不可能であった異方性化とバルク化を同時に実現することができる。これにより、R₂Fe₁₄B組成より高Fe側で、R₂Fe₁₄B相のバルク磁気特性を超える値を得ることが可能となった。

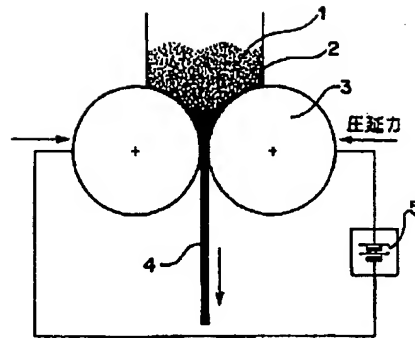
【図面の簡単な説明】

【図1】通電粉末圧延法の概念図である。

【符号の説明】

- | | |
|--------|-------|
| 1 粉末 | 4 シート |
| 2 ホッパー | 5 電源 |
| 3 ロール | |

【図1】



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CLAIMS

[Claim(s)]

[Claim 1] The rare earth, the iron, and the boron system magnet with which it consists of R (one or more sorts of the rare earth elements containing Y), Fe, and B, and the ratio of Fe is the nano composite magnet $R_2Fe_{14}B$ and whose soft phase are Fe or Fe_3B , and a hard phase is characterized by anisotropy-izing directly and being obtained from the quenching thin band of rare earth, iron, and a boron system alloy under existence of the liquid phase by 1 shaft deformation between ** above 82 atom %.

[Claim 2] The ratio of Co more than 82 atom % within 20 atom % of the sum total of Fe and Co [the ratio which consisted of R (one or more sorts of the rare earth elements containing Y), Fe, Co, and B, and Fe and Co totaled] It is the nano composite magnet whose hard phase is $R_2(Fe-Co)_{14}B$ and whose soft phase is Fe-Co or $(Fe-Co)_3B$. According to 1 shaft deformation between ** from the quenching thin band of rare earth, iron, and a boron system alloy under existence of the liquid phase The rare earth, the iron, and the boron system magnet directly characterized by anisotropy-izing and being obtained.

[Claim 3] R (one or more sorts of the rare earth elements containing Y), Fe, and M (aluminum, V, and Mo --) By one or more sorts of Zr, Ti, Sn, Cu, and Ga, and the atomic percentage, less than 4%, A hard phase is the nano composite magnet $R_2Fe_{14}B$ and whose soft phase it consists of B and the ratio of Fe is Fe or Fe_3B fundamentally more than 82 atom %. According to 1 shaft deformation between ** from the quenching thin band of rare earth, iron, and a boron system alloy The rare earth, the iron, and the boron system magnet characterized by anisotropy-izing directly and being obtained under existence of the liquid phase.

[Claim 4] R (one or more sorts of the rare earth elements containing Y), Fe, Co, and M (aluminum and V --) The ratio of Co more than 82 atom % less than 4% within 20 atom % of the sum total of Fe and Co [the ratio

which consisted of B by one or more sorts of Mo, Zr, Ti, Sn, Cu, and Ga, and the atomic percentage, and Fe and Co totaled] Or (Fe-Co) (Fe-Co) it is the nano composite magnet which is 3B. fundamental -- a hard phase -- R2(Fe-Co) 14B and a software phase -- under existence of the liquid phase according to 1 shaft deformation between ** from the quenching thin band of rare earth, iron, and a boron system alloy The rare earth, the iron, and the boron system magnet directly characterized by anisotropy-izing and being obtained.

[Claim 5] The manufacture approach of of the rare earth, the iron, and the boron system magnet according to claim 1 to 4 which carries out 1 shaft deformation of the quenching thin band of rare earth, iron, and a boron system alloy between ** under existence of the liquid phase, and is directly characterized by anisotropy-izing.

[Claim 6] The manufacture approach of of the rare earth, the iron, and the boron system magnet according to claim 5 characterized by removing the liquid phase condensed by the magnet alloy periphery section after **** 1 shaft deformation.

[Claim 7] The manufacture approach of of the rare earth, the iron, and the boron system magnet according to claim 5 characterized by performing temperature up to retention temperature within 5 minutes 2 seconds or more, and performing the temperature fall from retention temperature to 300 degrees C or less within 10 minutes 5 seconds or more in a **** 1 shaft deformation process.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] When this invention is used for electronic

equipment, especially the actuator for head actuation of a hard disk drive, it relates to optimal rare earth, iron and boron system magnet, and its manufacture approach.

[0002]

[Description of the Prior Art] Since the neodymium, the iron, and the boron magnet which makes $\text{Nd}_2\text{Fe}_{14}\text{B}$ the main phase were discovered by Sakawa, Croat and others in recent years, neodymium, iron, and a boron system magnet have made the magnetic properties improve by development of the manufacture approach which pulls out its presentation, and optimization and the high magnetic properties of an additive, amelioration, etc. (BH) max equivalent to about 88% of the theoretical value of maximum energy product (BH) max is realized by combination, such as formation of formation of low Nd high Fe presentation (in addition, atomic percentage of $\text{Nd}_2\text{Fe}_{14}\text{B}$ compound is Nd and 5.9%B and 82.3%Fe 11.8%) and bottom acid-ized process, high magnetic field orientation, and organization detailed. [of bringing the neodymium, the iron, and the boron system sintered magnet made with powder-metallurgy processing among the various manufacture approaches close to a stoichiometric ratio] However, an improvement of the magnetic properties by the process of powder-metallurgy processing, amelioration of a presentation, etc. is also reaching a limitation. Specifically with powder-metallurgy processing, the neodymium, the iron, and the boron system magnet with Fe presentation (more than 82%Fe) exceeding a stoichiometric ratio presentation of high performance cannot be obtained. It is because Fe phase will arise inevitably, this Fe phase that has software magnetism will cause flux reversal and coercive force will no longer be acquired, if that reason becomes a high Fe presentation. moreover, a stoichiometric ratio -- Nd -- it is because it is thought that it has contributed to coercive force generating of the generation length mold from a nucleus by the low-melt point point Nd rich phase which exists by rich presentation (that is, Fe PUA presentation) turning into the liquid phase in a sintering process, and cleaning the front face of a $\text{Nd}_2\text{Fe}_{14}\text{B}$ particle.

[0003] As the manufacture approach of anisotropy neodymium, iron, and a boron system magnet, the 1 shaft displacement method between ** is known in addition to powder-metallurgy processing. This approach bulk-izes with a hotpress the quenching thin band (a trade name MQ1, product made from MQI) which is a microcrystal thin band of the neodymium, iron, and boron obtained by heat treatment of an amorphous thin band or control of a cooling rate. It is the approach of obtaining the anisotropy neodymium, the iron, and the boron magnet (a trade name MQ3, product made from MQI)

in which the easy magnetization shaft carried out orientation in the application-of-pressure direction, by considering as a bulk isotropy magnet (a trade name MQ2, product made from MQI), and carrying out 1 shaft deformation of this bulk isotropy magnet between **. Extent of the anisotropy of the neodymium, the iron, and the boron magnet obtained by the **** 1 shaft displacement method has extent of hot-press deformation, and correlation, and the degree of an anisotropy also becomes large, so that whenever [deformation] becomes large. And (BH) max equivalent to about 75% of the theoretical value of (BH) max is realized by this approach. however, the 1 shaft displacement method between ** -- Nd -- since deformation takes place only by the presentation in which a rich low melting point phase (1 shaft deformation process between ** liquid phase) exists, in the stoichiometric ratio presentation in which a low melting point phase does not exist, and higher Fe presentation, it is inapplicable. Therefore, in the conventional 1 shaft displacement method between **, it was difficult to raise magnetic properties rather than the neodymium, the iron, and the boron system magnet obtained with powder-metallurgy processing.

[0004] As for the nano composite magnet with which a software phase and a hard phase form a detailed organization (10nm order), the software phase and the hard phase are unified by switched connection. And it is proved by both simulation and location survey that this nano composite magnet shows a permanent magnet property although the software phase exists. Therefore, if the high ingredient of saturation magnetization is used for the above-mentioned software phase, it may have high saturation magnetization and sufficient coercive force, and the high magnetic properties exceeding a hard phase may be shown. With a nano composite magnet, the necessity limited to specific combination does not have software phases (Fe, FeCo, Fe₃B-FeN system compound, etc.) and a hard phase (Nd₂Fe₁₄B, SmCo₅, Sm₂Co₁₇, Sm₂Fe₁₇Nx, NdTiFe₁₁Nx, other nitrides), and they can combine the thing in a parenthesis freely, and are not always limited by the presentation of a hard phase.

[0005] However, although the switched connection between particles can exist only in the detailed organization of effective 10nm order, anisotropy-izing such a detailed organization has not realized the nano composite magnet. Although the description of a nano composite magnet is in the point which shows comparatively high Br (residual magnetic flux density) when a software phase exists even if it is isotropic texture, it cannot discover sufficient coercive force and high (BH) max in isotropic texture.

[0006] Moreover, there is also a trouble that-izing cannot be carried

out [bulk] in a nano composite magnet. The approach of using as a bulk magnet, without hypertrophying the nano composite organization by which it was got, although a nano composite magnet is usually produced by the melt quenching method, the mechanical alloying method, etc. and is obtained in the form of powder or a thin band is not devised yet. The special method of making fine particles bulk-size according to the pulse extra-high voltage force is not the approach suitable for utilization of a certain thing at all. As mentioned above, in the nano composite magnet, it was impossible to have realized anisotropy-izing and bulk-ization simultaneously.

[0007]

[Problem(s) to be Solved by the Invention] Then, this invention is the nano composite magnet which made $R_2Fe_{14}B$ the hard phase, and aims at offering anisotropy-izing, the rare earth, the iron and the boron system magnet which were bulk-ized, and its manufacture approach.

[0008]

[Means for Solving the Problem] This invention R (one or more sorts of the rare earth elements containing Y), Fe (or Fe permuted by Co of the specified quantity), It consists of M (it is less than 4% at one or more sorts of aluminum, V, Mo, Zr, Ti, Sn, Cu, and Ga, and an atomic percentage) as occasion demands further. B -- the ratio of Fe above 82 atom % It is the nano composite magnet whose hard phase is $R_2Fe_{14}B$ and whose soft phase is Fe or Fe_3B , and they are the rare earth, the iron, and the boron system magnet characterized by anisotropy-izing directly and being obtained from the quenching thin band of rare earth, iron, and a boron system alloy under existence of the liquid phase according to 1 shaft deformation between **. Another this invention is the manufacture approach of of the above-mentioned rare earth, iron, and boron system magnet which carries out 1 shaft deformation of the quenching thin band of rare earth, iron, and a boron system alloy between ** under existence of the liquid phase, and is directly characterized by anisotropy-izing. In this case, as for the liquid phase, it is desirable to consist of low melting alloys of the La-Fe system which has wettability to a hard phase, or a R-Cu system. It is good to remove the liquid phase condensed by the magnet alloy periphery section after 1 shaft deformation between ** in this invention, to perform temperature up to retention temperature within 5 minutes 2 seconds or more in 1 shaft deformation process between **, and to perform the temperature fall from retention temperature to 300 degrees C or less within 10 minutes 5 seconds or more.

[0009]

[Embodiment of the Invention] It makes $R_2Fe_{14}B$ a hard phase, and the rare earth, the iron, and the boron system magnet of this invention carry out switched connection between software phases with saturation magnetization higher than this hard phase, and it is anisotropy-izing and the bulk-ized nano composite magnet, and it is obtained in 1 shaft deformation process between ** by the presentation in which the liquid phase exists according to the process of 1 shaft deformation between direct ** from a quenching thin band, and rapid temperature up and forced cooling.

[0010] The rare earth, the iron, and the boron system magnet of this invention R (one or more sorts of the rare earth elements containing Y), It consists of M (one or more sorts of aluminum, V, Mo, Zr, Ti, Sn, Cu, and Ga) as occasion demands further. Fe (or Fe-Co) and B -- the ratio of Fe (or Fe-Co) It is a nano composite magnet with the presentation more than rich 82 atom %. the stoichiometric ratio of $R_2Fe_{14}B$ -- Fe -- In the hard phase, $R_2Fe_{14}B$ (or $R_2(Fe-Co)_{14}B$) and a software phase consist of Fe (or Fe-Co) or Fe_3B (or $(Fe-Co)_3B$). Although it is the rare earth elements containing Y, R is effective, if Tb and Dy are used for a part of R, in order to make Nd and Pr into a subject and to increase coercive force. Moreover, although Above Fe is replaceable at Co, since the magnetic properties of a hard phase fall by Co permutation, it is desirable [the permutation ratio] to make 20% of the sum total of Fe and Co into an upper limit by the atomic percentage. furthermore, Fe or Fe-Co -- an alloying element M (one or more sorts of aluminum, V, Mo, Zr, Ti, Sn, Cu, and Ga) -- small quantity -- you may permute. Since magnetic properties will fall too much when extent of the permutation by M becomes 4% or more to a total presentation by the atomic percentage although M has effectiveness in detailed-ization of an organization and it contributes to coercive force buildup, considering as less than 4% is desirable. However, it is better to add 0.1% or more preferably, since the effectiveness of detailed-izing stops showing up when there are too few additions of M. In addition, although it is so desirable that it is few about the amount of the impurity of C and N which are mixed at production of rare earth, iron, and a boron system alloy, or the process of magnet manufacture, and O and others, considering as less than 1% is unescapable.

[0011]. The rare earth, the iron, and the boron system magnet of this invention are manufactured from the thin band or powder (these are hereafter called quenching thin band) obtained by the melt quenching method or the mechanical alloying method. Although in either the detailed organization condition of 10nm order which switched connection

produced between particles, or an amorphous condition is sufficient as a quenching thin band, the amorphous condition is more desirable in order to control hypertrophy of the particle size of a magnet alloy particle as much as possible by the 1 shaft transformation path between **.

[0012] The rare earth, the iron, and the boron system magnet of this invention are obtained by carrying out 1 shaft deformation between **, and anisotropy-izing a quenching thin band directly. In the conventional approach, when carrying out 1 shaft deformation between ** and anisotropy-izing a quenching thin band, the quenching thin band was once bulk-ized with the hotpress, after carrying out temperature up of the acquired isotropic magnet block once again, 1 shaft deformation between ** was carried out, and it was anisotropy-ized. However, in this invention, rapid temperature up of the quenching thin band is carried out, and 1 shaft deformation between ** is carried out directly. Therefore, since only necessary minimum heat treatment is performed to a quenching thin band in an anisotropy-ized process, the hypertrophy of a detailed organization in 1 shaft deformation process between ** will be controlled, consequently switched connection between particles will fully be performed.

[0013] Above 82 atom %, as [Description of the Prior Art] described, deformation hardly takes place and the ratio of Fe does not anisotropy-ize the rare earth, the iron, and the boron system alloy whose hard phase is $R_2Fe_{14}B$, even if it performs 1 shaft application of pressure between **. Then, in order to solve this problem, by this invention, the ratio of Fe is more than 82 atom %, and rare earth, iron, and a boron system magnet alloy are prepared so that the liquid phase may exist by the 1 shaft transformation path between **. What this liquid phase turns into the liquid phase in 1 shaft deformation process between ** since wettability with rare earth, iron, and a boron system magnet alloy is bad and does not contribute to 1 shaft deformation between ** in rare earth, iron and a boron system magnet alloy, and the thing (for example, low-melt point point solder alloy) that is completely irrelevant, and has rare earth, iron and a boron system magnet alloy, and wettability is needed. this invention persons found out that a La-Fe system alloy or a R-Cu system alloy was desirable, as a result of examining many things about the alloy which fulfills such conditions. In order to obtain such an organization, La and Cu are directly added during an organization, or a La-Fe system alloy and a R-Cu system alloy are combined with a R-Fe-B system alloy. In this case, as for the addition of La and Cu, it is desirable to make it less than [2wt%] to the original presentation. In addition, the binary alloy of a La-Fe system does not make an

intermetallic compound, and the ternary alloy of a La-Fe-B system cannot generate $\text{La}_2\text{Fe}_{14}\text{B}$ very easily. The binary alloy of a La-Fe system serves as the liquid phase below 800 degrees C by the eutectic reaction with La rich side. By there being rare earth, iron and an original boron system magnet alloy, and original similarity, since this liquid phase has good wettability with $\text{R}_2\text{Fe}_{14}\text{B}$ phase, it contributes to 1 shaft deformation between **. Since the compound with which Cu does not permute Fe of $\text{R}_2\text{Fe}_{14}\text{B}$, and one R-Cu system alloy is also generated by the R-Cu system has the low melting point, it is suitable as the liquid phase of this invention. In this invention, such a low melting point phase turns into the liquid phase in 1 shaft deformation process between **, and difficult anisotropy-ization is attained by the conventional technique by performing reorientation through this liquid phase.

[0014] After fully carrying out application-of-pressure deformation and anisotropy-izing a quenching thin band directly according to 1 shaft deformation through the liquid phase between **, the liquid phase which consists of low melting alloys of a La-Fe system (or R-Cu system) is pressed out and condensed by the magnet alloy periphery section which becomes free [a pressure]. A liquid phase component will be in the condition of hardly existing in a magnet alloy core, in that case. By the approach of anisotropy-izing like the conventional approach, after using a magnet alloy as an isotropic bulk magnet, a low-melt point point liquid phase component is not condensed in the magnet alloy periphery section, but is distributed to homogeneity inside. That is, like this invention, the phenomenon in which the low-melt point point liquid phase is condensed in the magnet alloy periphery section is seen, only when 1 shaft deformation of the quenching thin band between ** is carried out directly. Since the low-melt point point liquid phase is nonmagnetic, after not contributing to the magnetic properties of a permanent magnet but completing 1 shaft deformation between **, it is only reducing magnetic properties. Therefore, the anisotropy nano composite magnet which has high magnetic properties is obtained by removing the magnet alloy periphery section by which the low-melt point point liquid phase was condensed, and using the core which almost consists of a magnetic phase.

[0015] Since hypertrophy of a very fine particle will take place if 1 shaft deformation between ** of a quenching thin band is not performed for a short time, in order to avoid this, in 1 shaft deformation process between **, it is desirable to perform temperature up to the retention temperature which carries out 1 shaft deformation between ** within 5 minutes 2 seconds or more, and to perform the temperature fall from

retention temperature to 300 degrees C or less within 10 minutes 5 seconds or more. In this case, retention temperature is 500-1000 degrees C. There is energization powder rolling as an example of the concrete approach in which the high-speed temperature up of such a short time and a temperature fall are possible. Since a roll outlet will serve as a maximum temperature if the powder 1 of the quenching thin band by machine grinding is thrown in in a roll 3 from the hopper 2 upper part as shown in drawing 1 , and a high current is energized through the powder rolled out, this approach is an approach of fabricating on a sheet 4 and performing deformation between 1 ****, by pressurizing powder 1 with this roll 3. When pressurizing according to this approach, a direction parallel to the shaft of a roll 3 serves as 1 axial compression, in order that a pressure may escape. Moreover, since the quenching thin band is powdered, even if it energizes, a current does not flow, therefore temperature up hardly arises from a power source 5, until compression with a roll 3 starts. That is, in order to stop energizing when it energizes for the first time and rolled stock comes out of a roll 3 by being compressed to some extent into a roll 3, and to enter at a temperature fall phase, the time amount by which the quenching thin band is heated is a short time very much.

[0016] According to this energization powder rolling, by the time it becomes the organization which crystallized from the amorphous condition (or fine crystal condition), the particle size of a magnet alloy will seldom get fat, but an anisotropy bulk-sized nano composite magnet with the detailed organization of 10nm order is obtained. The maximum temperature which performs **** 1 shaft deformation, and the rate of temperature up and a temperature fall can adjust the current value and roll 3 rotational frequency which are energized between rolls 3, and an application-of-pressure deformation degree can be optimized by adjusting the pressure and spacing during a roll. As for the energization rolling section, it is desirable that they are an inert gas ambient atmosphere or a vacuum ambient atmosphere in order to prevent the oxidation degradation of a rolling object. One step or multistage are available for a roll 3. In addition, as long as the method of performing 1 shaft deformation between ** is an approach of it not being limited to the above-mentioned approach and having the same functions, such as an application-of-pressure spark sintering method, any are sufficient as it.

[0017]

[Example] Hereafter, although the example of this invention is shown, this invention is not limited to this.

(Example 1) Liquid quenching was performed for Nd, 1%La, 76%Fe, and the

dissolution alloy that consists of Co and 5%B and an unescapable impurity 10% under the reduced pressure Ar gas ambient atmosphere 8% by the atomic percentage, and it considered as the amorphous quenching thin band with the single roll equipment of peripheral-speed 60 m/sec. Next, this amorphous quenching thin band was ground by machine, it considered as the powder of 100 or less meshes, and powdered anisotropy-izing and the formation of bulk sheet metal which were obtained by energization powder rolling within Ar gas ambient atmosphere were performed simultaneously. 1 axial stress is 500kg/cm² on an average in that case. The current was 10kA and roll peripheral speed was 1 mm/sec, and temperature up was carried out from the room temperature in about 20 seconds to the retention temperature field (800 degrees C) which carries out 1 shaft deformation between **, and the temperature was lowered to 300 degrees C or less in about 40 seconds. The great portion of La is condensed by the edge among the sheet metal of 1mm thickness by produced 20mm width of face, 2.5mm of ends which are these parts was removed, and the magnetic properties which are Br=1.66T and iHc=800 kA/m and anisotropy-ized the sheet metal of remaining 15mm width of face when Br and iHc were measured in the application-of-pressure direction were acquired. Moreover, the presentation of the obtained sheet metal is Nd, 0.1%La, 76.5%Fe, 10.1%Co, and 5.1%B 8.1%, and consisted of Fe-Co and Nd₂(Fe-Co)₁₄B.

[0018] (Example 2) By the atomic percentage, Pr, 1.5%La, and the dissolution alloy that consists of Fe and 5%B and an unescapable impurity 87.5% were quenched on the same conditions as an example 1 6%, and the amorphous quenching thin band was produced. Moreover, 2.5mm of ends which are the parts of La which considered as bulk sheet metal by the energization powder rolling of the same conditions as these conditions, and was condensed by the edge among the sheet metal of 1mm thickness by produced 20mm width of face was removed, and the magnetic properties which are Br=1.53T and iHc=990 kA/m and anisotropy-ized the sheet metal of remaining 15mm width of face when Br and iHc were measured in the application-of-pressure direction (the direction of 1mm thickness) were acquired. Moreover, the presentation of the obtained sheet metal is 6.5%Pr, 0.1%La, 88%Fe, and 5.4%B, and consisted of Fe and Pr₂Fe₁₄B.

[0019] (Example 3) 5% was mixed [the thing which consists of Nd, 82.5%Fe, and 8%B, 1%aluminum, 0.5%Ti, and an unescapable impurity 8% by the atomic percentage and which was alloyed beforehand] with 95% for the SmCu alloy to the predetermined ratio by the weight ratio by the weight ratio, liquid quenching was performed under the reduced pressure

Ar gas ambient atmosphere, and this mixed dissolution alloy was used as the amorphous quenching thin band with the single roll equipment of peripheral-speed 60 m/sec. Next, except having set the current value of energization powder rolling to 15kA, when Cu condensed at the edge like the example 1 was removed, sheet metal was produced and Br and iHc were measured in the application-of-pressure direction, it is Br=1.45T and iHc=1250kA/m and the anisotropy-ized magnetic properties were acquired. When the powder of 20 or less meshes which carried out manual grinding of the obtained sheet metal was measured by the X diffraction, it has checked consisting of Nd₂Fe₁₄B and Fe₃B fundamentally.

[0020] (Example 4) Liquid quenching was performed for Nd, 1%La, 84.5%Fe, and 5%B and the dissolution alloy which consists of Cu, 0.5%Mo, and an unescapable impurity 1% under the reduced pressure Ar gas ambient atmosphere 8% by the atomic percentage, and it considered as the amorphous quenching thin band with the single roll equipment of peripheral-speed 60 m/sec. Next, except having set the current value of energization powder rolling to 15kA, when Cu condensed at the edge like the example 1 was removed, sheet metal was produced and Br and iHc were measured in the application-of-pressure direction, it is Br=1.53T and iHc=1040kA/m and the anisotropy-ized magnetic properties were acquired. When the powder of 20 or less meshes which carried out manual grinding of the obtained sheet metal was measured by the X diffraction, it has checked consisting of Nd₂Fe₁₄B and Fe₃B fundamentally.

[0021]

[Effect of the Invention] Impossible anisotropy-izing and bulk-ization are simultaneously realizable with a nano composite magnet with this invention until now by carrying out 1 shaft deformation of rare earth, iron, and the boron system magnet alloy between ** directly on high-speed temperature up and high-speed temperature fall conditions under existence of the liquid phase. This became possible from the R₂Fe₁₄B presentation to acquire the value exceeding the bulk magnetic properties of R₂Fe₁₄ B phase by the high Fe side.

[Translation done.]

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the conceptual diagram of energization powder rolling.

[Description of Notations]

- 1 Powder 4 Sheet
- 2 Hopper 5 Power Source
- 3 Roll

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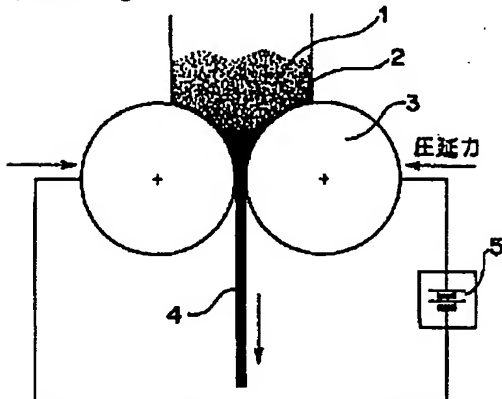
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DRAWINGS

[Drawing 1]



[Translation done.]